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Influence of Sodium Carbonate upon the Producer Gas Reaction

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Fox and White have shown that the reaction $Na_2CO_3 + 2C = 2Na + 3CO$ *proceeds to the right at temperatures above 800° C., and that the vapor pressure of the reaction products surpasses atmospheric pressure about 1025° C. In their work the reaction products were cooled rapidly to prevent reversal of the reaction. In the present study the reaction products are allowed to cool more slowly, as would be the case in a gas producer, and it is found that reversal is rapid and complete in the range 750° to 900° C. The study of the application of this reaction to gas producer practice is made in a miniature gas producer consisting of a nickel tube inserted in an electric furnace. When using untreated Acheson graphite and dry air at 900° C. and a time of contact of 2 seconds, the exit gases contain 6.6 per cent carbon monoxide; under similar conditions using graphite impregnated with sodium carbonate, 33.4 per cent carbon monoxide is found. As little as 0.1 per cent sodium carbonate is sufficient to give this result. The decomposition of moistened air is accelerated in a similar way. The sodium carbonate is decomposed in the lower part of the producer where the temperature is high, and is regenerated in the upper and cooler part of the producer where it deposits upon the graphite and is carried down*

mechanically to the reaction zone. By using soda, a satisfactory gas composition is obtained at a maximum temperature of 925° C., while without soda satisfactory operation is not possible even at 1040° C. Part of the soda is carried out of the producer with the fuel ash, and the extent of this loss has not been sufficiently studied. If it were not for this loss, a small amount of soda would last a long time, since it is continuously recycling within the producer.

THE effect of sodium carbonate in promoting the rate of the reaction of carbon dioxide has been ascribed by Dent and Cobb (*1*) to an alteration of the nature of the carbon surface exposed to the gas. Fox and White (*2*) explained the apparent increase in the reactivity of graphite which was impregnated with sodium carbonate as being due to a reaction between the carbon and sodium carbonate which was measurable at temperatures above 750° C. and which evolved a continuous stream of gaseous products at temperatures above 1025° C. according to the equation:

$$2C + Na_2CO_3 \rightleftharpoons 2Na + 3CO$$

They held that the vapors of sodium and the carbon monoxide evolved at the surface of the carbon ruptured the stagnant gas film at the surface of each particle and permitted more ready reaction between the carbon and carbon dioxide. Furthermore, the sodium vapor, after getting out into the main stream of gas, reacted with a molecule of carbon dioxide to form sodium oxide and carbon monoxide, and the sodium oxide reacted with a second molecule of carbon dioxide to regenerate the sodium carbonate. Neumann, Kröger, and Fingas (*3*) reported that potassium carbonate behaved in a similar manner, and they gave a similar explanation of its behavior.

Fox and White cooled the reaction products so quickly that reversal was prevented. The temperature of reversal of this reaction formed the first part of the present study. It was made in an electrically heated nickel tube placed in a furnace with two separate heating elements. The lower half of the tube contained granules of Acheson graphite impregnated with sodium carbonate and was maintained at 1050° C. The upper half was empty and was kept at 750° C. When the furnace was up to heat, the reversal within the furnace was so complete that only enough carbon monoxide came out of the small aperture at the top of the tube to give a flame the size of a pinhead. On removing the nickel tube and cooling rapidly, a hard black incrustation of sodium carbonate and carbon was found in the upper half of the tube. The reversal had been almost quantitative in the region where the temperature dropped from 900° to 750° C.

A laboratory study of the effect of the addition of sodium carbonate to graphite on the producer gas reaction was made in an electrically heated furnace containing a nickel tube 45 mm. in diameter and 350 mm. long, which had welded into its upper end a nickel tube 20 mm. in diameter and of the same length. A smaller inlet tube permitted air to be passed into the bottom, and a pyrometer well extending from the top of the tube permitted internal temperatures to be measured at various levels. External temperatures were measured by a thermocouple in the space between the nickel tube and the alundum tube on which the windings were supported. In commencing a test the current was turned on, and a metered volume of air was started up through the graphite when the inside thermocouple showed a temperature of 400° C. After a preliminary run of 20 minutes the inside thermocouple was

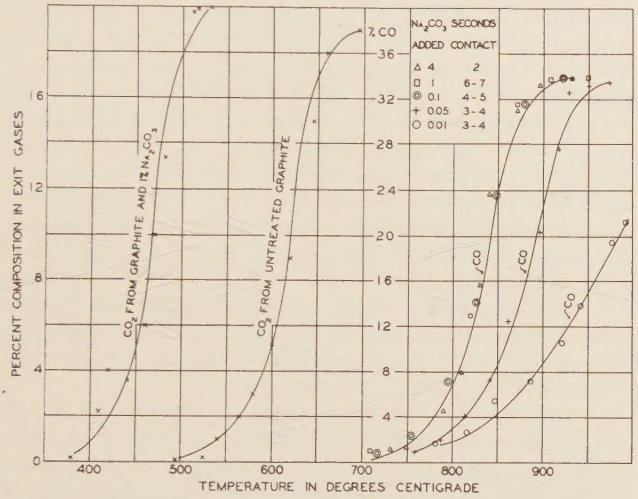

FIGURE 1. INFLUENCE OF SODIUM CARBONATE ON COMPOSITION OF GASES EVOLVED FROM INTERACTION OF GRAPHITE AND DRY AIR

placed at that point which showed the maximum temperature, and the outside couple was placed opposite this point. Meter readings of the inlet air and analyses of the gases leaving the furnace were made at intervals as the temperature rose. The air was sometimes dried with calcium chloride and sometimes humidified to a definite degree. The temperatures recorded in the graphs are the arithmetical average of the maximum temperature in the center of the tube and that of the thermocouple on the outside of the reaction tube, which was 40° to 70° C. higher than the center thermocouple. The recorded temperature is therefore somewhat hotter than the mean temperature in the zone of highest temperature. The time of contact was calculated from the volume of entering air and steam when heated to the recorded temperature without taking account

of increase in volume due to formation of carbon monoxide and hydrogen. Separate reaction tubes were used for the untreated graphite and that treated with soda so that there might not be any accidental contamination of the untreated graphite. The experimental methods were the same for treated and untreated graphite so that the sets of experiments are strictly comparable.

Effect of Varying Quantity and Method of Adding Sodium Carbonate

Since sodium carbonate is a solid below 850° C. and a liquid above that point, it is evident that intimate mixture of the two substances must be provided in order that reaction may be rapid. The most effective method is to soak the graphite in a solution of sodium carbonate and then dry the granules. Experiment showed this procedure was distinctly more effective at temperatures above 800° C. than merely mixing the dry soda and graphite together. However at lower temperatures where the evolution of sodium is not measurable, the coating of solid sodium carbonate deposited on the graphite granules hindered the reaction between the graphite and oxygen. At 750° C. the gas from the coated graphite showed only 3 per cent carbon monoxide while that from the graphite mixed with soda showed 6 per cent carbon monoxide. However the reaction between graphite and oxygen to form carbon dioxide was accelerated in the temperature range of 400° to 500° C. by impregnation with soda as shown in Figure 1, where at 500° C. the untreated graphite showed scarcely any reactivity while that impregnated with sodium carbonate showed 18 per cent carbon dioxide. This is discussed later.

The effect of varying percentages of soda on the rate of reaction between graphite and dry air is given graphically in Figure 1, where curves show the influence of percentages of soda varying from 0.01 to 4.0 per cent. The time of contact varies from 2 to 7 seconds. The curves for 0.1, 1.0, and 4.0 per cent of sodium carbonate are practically coincident and show almost equilibrium conditions with 33 per cent carbon monoxide at 900° C. Impregnation with 0.05 and 0.01 per cent sodium carbonate was less effective. Although it thus appears that impregnation with 0.1 per cent sodium carbonate is adequate to give maximum results under the present experimental conditions, all subsequent tests were run with material containing 1.0 per cent sodium carbonate unless expressly stated.

Influence of Time of Contact

The effect of shorter times of contact on the reaction between dry air and graphite with one per cent and without any sodium carbonate is shown graphically in Figure 2 where the percentages of carbon monoxide are plotted against temperature for various reaction periods. It is evident that the effect of one per cent of soda has been to increase the apparent

reactivity of the graphite so that better results are obtained with the treated graphite than are obtainable with the untreated graphite at a temperature higher by 200° C. With a 2-second contact the gas from the treated graphite contains 31 per cent carbon monoxide at 850° C. while that from the untreated graphite contains only 25 per cent at 1050° C. With the treated graphite a gas with 33.2 per cent carbon monoxide is formed in 0.5 second at 950° C.; under similar conditions with untreated graphite the gas contains only 5.8 per cent carbon monoxide. With a 2-second contact the treated graphite gives 33.6 per cent carbon monoxide at 900° C. and the untreated only 6.8 per cent carbon monoxide. The curves for longer contacts, 4 and 7 seconds, respectively, show an apparent anomaly in that the carbon

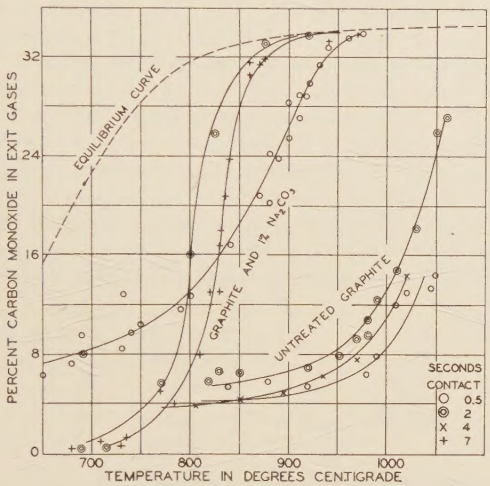

FIGURE 2. INFLUENCE OF TIME OF CONTACT ON COMPOSITION OF GASES EVOLVED FROM INTERACTION OF UNTREATED GRAPHITE AND DRY AIR AS COMPARED WITH GRAPHITE IMPREGNATED WITH ONE PER CENT SODIUM CARBONATE

monoxide percentage is lower than in that for 2 seconds. This is probably due to the reversal of the reaction,

$$2CO \rightleftharpoons C + CO_2$$

in the longer time given for cooling the gases with the slower rate of air passing through the apparatus. Another apparent anomaly is the higher percentage of carbon monoxide in the case of treated graphite with 0.5-second contact at temperatures below 800° C. At this very rapid rate of blowing, with the large proportion of the carbon oxidized to carbon dioxide and a large amount of heat evolved, the hot zone in the producer probably broadened and gave a gas relatively higher in carbon monoxide than that obtained when heat was being evolved less rapidly and the temperature of the

fuel bed was still not high enough to cause much reduction of carbon dioxide to carbon monoxide.

Behavior of Air-Steam Mixtures

In the work with dry air the cold air impinged directly on the grate supporting the fuel. When using steam the grate was raised within the furnace so that the air-steam mixture was preheated to about 800° C. before it met the graphite. Temperatures were measured at the grate level, at the level of the middle of the fuel column both inside and outside of the producer, and at the point above the fuel bed where the gases entered the smaller off-take pipe. The calculation of the time of contact was based upon the inlet volume of air and steam when raised to the average temperature at the middle of the fuel column. The times of contact were all short, varying from 0.3 to 1 second. The amount of steam per pound of air varied from 0.087 to 0.198 pound.

The results of a run in which 0.119 pound of steam per pound of air was used are shown graphically in Figure 3. The time of contact in this series was 0.5 second for the impregnated and 0.6 second for the untreated graphite. In general the effect of the soda was to increase the apparent reactivity of the graphite so that similar results were obtained at a temperature 150° C. lower than when no soda was used. The following comparisons are taken from the curve:

	900° C., 1% Na_2CO_3	1050° C., No Na_2CO_3
CO, %	30	28
H_2, %	11	9
CO_2, %	1	5
H_2O decomposed, %	86	65

These curves of Figure 3 are rather similar to some given by Fox and White (*2*) and those for Neumann using potassium carbonate. The results of the series of tests with other proportions of steam show results similar to those of Figure 3 and are not reported because of lack of space.

Fox and White (*2*) explained this increase in reactivity by the alternate reduction of sodium carbonate and reoxidation of the products to reform sodium carbonate. Neumann, Kröger, and Fingas (*3*) gave the same explanation for graphite treated with potassium carbonate. According to this work their theories are logical. The reaction of sodium carbonate with carbon of the fuel bed gives products that rupture the film of carbon monoxide around each graphite granule, making it possible for water and carbon dioxide to react with fresh carbon surfaces. The products of the reaction (sodium or sodium oxide) travel up the producer with the gases, reacting with any carbon dioxide or water that may remain. The main portion of this secondary reaction occurs in the cooler or upper end of the gas producer, forming sodium carbonate again. Most of this regenerated carbonate is filtered

out by the surrounding graphite and, along with it, works its way back down into the reduction zone.

Further experimental work on this regeneration of sodium carbonate was carried out by operating the miniature producer already described with treated graphite and then replacing the graphite as it burned away by untreated graphite added at the top of the column. The graphite initially added carried 0.6 per cent sodium carbonate; after 7 hours of operation, when the charge consisted of almost 100 per cent of untreated graphite, the percentage composition of the gas was carbon dioxide 5.7, carbon monoxide 28.0, hydrogen 11.4. It had a heating value of 127 B. t. u. per cubic foot, whereas

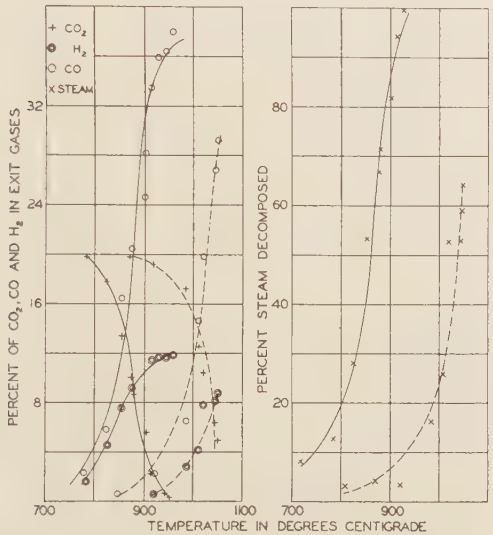

FIGURE 3. EFFECT OF SODIUM CARBONATE IN ACCELERATING RATE OF REACTION OF STEAM AND AIR UPON GRAPHITE

Broken lines, gas from untreated graphite; full lines, composition of gas from graphite impregnated with one per cent sodium carbonate.

if untreated graphite had been used through the run, the heating value of the gas would have been only 49 B. t. u. per cubic foot.

In a test designed to determine what changes took place in the soda during a run, the producer was charged with graphite containing 1.7 per cent sodium carbonate. The upper constricted part of the producer was filled with untreated graphite. This wedged in the tube so that it did not feed down into the reaction zone but acted merely as a filter. This test was made at an operating temperature of 950° C. and with a gas composition which averaged 0.5 per cent carbon dioxide, 36.7 per cent carbon monoxide, and 12.2 per cent hydrogen for 6 hours. After that period the carbon

dioxide commenced to increase, and the run was discontinued after 7.5 hours when the carbon dioxide had risen to 3.8 per cent. When the apparatus was cooled and examined, it was found that the fuel bed, originally 12 inches (30.5 cm.) deep, had burned away to a depth of 2 inches (5 cm.). This shallow fuel bed with a time of contact of only 0.1 second was insufficient to cause complete reduction of the carbon dioxide and explained the increase in that constituent of the gas during the last hour of the test. The percentage of soda recovered in the various parts of the apparatus was determined separately with the following results:

	Na_2CO_3	
	Weight	Per cent by weight
	Grams	
In fuel in active zone	2.4	26.4
On walls of producer	1.4	15.4
Collected on graphite in cooler upper part of producer	4.2	46.2
In gas washers	0.04	0.4
Total recovered	8.04	88.4
Unaccounted for	1.06	11.6
	9.10	100.0

Part of the unaccounted for loss was due to spilling a solution, but part of the soda undoubtedly combined with the ash, which was not analyzed.

The only method by which the soda could have been transferred to the upper portion of the producer was by means of the rising gases, since the velocity of the gases in the reaction zone was less than that in the constricted zone where the soda was deposited. Sodium carbonate does not have an appreciable vapor pressure at the temperature of this experiment, and it must therefore have reacted to give products that were volatile. These might have been sodium, a vapor at the reaction temperature, or sodium oxide of whose properties little is known. If the fuel is free from ash, and no excess of soda is used, it may be expected that a quantitative recovery of the soda may be obtained.

Effect of Ash

The previous tests had been made on graphite which was selected because of its reproducibility. Experiments were then made on foundry coke which was impregnated with a solution of sodium carbonate and then dried. Coke impregnated with one per cent sodium carbonate showed hardly any effect due to the soda, and it was surmised that the ash of the coke was combining with the soda and fixing the sodium as a silicate. Coke was accordingly treated with 5.0 per cent of soda, and that amount gave the effect expected, as shown by the data of Table I. The treated coke gave a richer gas at 880° C. than could be obtained from the untreated coke at 1090° C.

No quantitative measurement has been made of the amount of soda carried out in combination with the ash.

TABLE I. EFFECT ON PRODUCER GAS REACTION OF 5 PER CENT SODIUM CARBONATE ADDED TO COKE

Av. Central Zone Temp. ° C.	CO_2 %	CO %	H_2 %
	TREATED COKE		
780	18.2	6.0	5.0
820	13.2	15.0	7.8
880	5.6	28.4	10.4
945	2.0	32.8	10.8
970	1.6	34.0	11.4
	UNTREATED COKE		
780	19.4	1.8	1.8
845	18.8	3.0	2.6
940	15.6	8.6	6.4
975	14.6	10.4	7.2
1090	9.8	19.8	8.6

DISCUSSION OF RESULTS

The increase in apparent reactivity of carbon at temperatures above 750° C. when treated with sodium carbonate, which has been shown by several investigators, has been corroborated by this work. The various investigators have offered conflicting explanations as to how sodium carbonate brings about this increase in apparent reactivity. Dent and Cobb (*1*) state that sodium carbonate influences the allotropic modification of the carbon that is exposed to the gases. Fox and White (*2*) suggest that the catalytic action of the soda is probably due to an alternate reduction and oxidation as shown by the following equations:

$$Na_2CO_3 + 2C \rightleftharpoons 2Na + 3CO$$
$$CO_2 + 2Na \rightleftharpoons Na_2O + CO$$
$$Na_2O + CO_2 \rightleftharpoons Na_2CO_3$$

Neumann (*3*) et al. suggest a similar explanation and give similar equations when potassium carbonate is substituted for sodium carbonate.

According to the present investigation the alternate reduction of sodium carbonate and oxidation of the products of the reaction is the best means by which the mechanism of the process can be explained. If the sodium carbonate affected only the allotropic modification of the carbon that was exposed to the gases, it would collect at the bottom of the producer when the carbon to which it had originally adhered had burned away. Just the opposite was found to be true. Although the percentage of soda on the graphite in the bottom of the producer did increase, the greatest amount of it had been transferred to the graphite at the top of the fuel bed. Since sodium carbonate is not appreciably volatile at temperatures in the producer (700° to 950° C.), it must have been reduced by carbon to sodium and carbon monoxide, which are volatile at these temperatures, and have been carried upward by the gases. In going upward with the gases, the sodium reacted with carbon dioxide, forming sodium oxide and carbon monoxide, and the sodium oxide further reacted with another molecule of carbon dioxide to form sodium carbonate. Some of the sodium also reacted with steam.

Below 800° C. the film of solid sodium carbonate around each graphite particle seems to retard the formation of carbon monoxide by decreasing the surface of graphite in direct contact with the gases. This same film of sodium carbonate apparently decreases the temperature at which the graphite and oxygen of the air react to form carbon dioxide, by about 150° C. All of the oxygen of the air is converted to carbon dioxide by reacting with the treated graphite at approximately 525° C.; under otherwise similar conditions 675° C. is required with a fuel bed of untreated graphite. A suggestion of the mechanism of this low-temperature reaction is:

$$Na_2CO_3 + C \rightleftharpoons Na_2O + 2CO$$
$$2CO + O_2 \rightleftharpoons 2CO_2$$
$$Na_2O + CO_2 \rightleftharpoons Na_2CO_3$$

Enough work has not been done to prove that these equations take place as written, although thermodynamic calculations indicate their probability at 600° to 700° C.

The amount of sodium carbonate needed to bring about the maximum increase in apparent reactivity of the graphite is very small, being less than one per cent. Since the products of decomposition of the carbonate tend to work their way up to the cooler regions of the producer, and the regenerated carbonate is then carried back down into the reducing zone by the fuel on which it was deposited, additions of more carbonate need be made only occasionally. Some of the carbonate that is lost is carried out in the ash, either by combining with the siliceous matter or being carried out as the free carbonate, and in rapid operation some may be carried out in the gas. These sources of loss require further investigation.

The length of active fuel bed in this investigation was generally held between 250 and 300 mm. It was found that a fuel bed only 50 mm. deep, containing approximately 3 per cent sodium carbonate, gave gases that were richer in carbon monoxide and hydrogen than those coming from fuel beds of untreated graphite whose active fuel bed was about 250 mm. deep, both being at the same temperature, and the velocity of gases through each being the same. Fuel beds of treated and untreated graphite of the same depths, of the same temperature, and having the same total time of contact of gases with fuel, show vast differences in exit gas composition. At 950° C. the percentage composition of gases from a treated fuel bed was: carbon dioxide 1, carbon monoxide 36, hydrogen 11.8, and nitrogen 51.2; from an untreated fuel at the same temperature: carbon dioxide 17, carbon monoxide 5, hydrogen 2, and nitrogen 76.0. At 1040° C. this same untreated fuel bed has an exit gas composition of 9 per cent carbon dioxide, 23 per cent carbon monoxide, 7.6 per cent hydrogen, and 60.4 per cent nitrogen.

Commercial Applicability of Process

A comparison of the heating values and the maximum theoretical flame temperatures of gases coming from a producer using a treated fuel and one using an untreated fuel will indicate some of the possibilities of this process.

Operating at a reducing zone temperature of 950° C., the average composition of the gases coming from a fuel bed of treated graphite would analyze, in per cent: carbon dioxide 1, carbon monoxide 35.4, hydrogen 9, and nitrogen 54.6, with approximately 100 per cent decomposition of water. The exit gas temperature would be about 800° C. The heating value of this gas is 143 B. t. u. per cubic foot. The maximum theoretical flame temperature, based upon the complete combustion of carbon monoxide and hydrogen at an initial temperature of 800° C. with no excess air and no radiation losses, is 2400° C. The average percentage analysis of the gas in commercial producer practice as given by Haslam and Russell is: carbon dioxide 5.7, carbon monoxide 22, hydrogen 10.5, and nitrogen 61.8, with 60 per cent water decomposed and an exit gas temperature of 800° C. Based upon the same conditions given above, the B. t. u. per cubic foot of this gas are 98, and the maximum theoretical flame temperature is 2100° C. If the gases are burned while hot, the treated fuel shows an advantage of having a maximum flame temperature that is 300° C. higher. If they are cooled and then used, the treated fuel shows an advantage of 45 B. t. u. per cubic foot and a cold gas efficiency of 84 per cent as compared to 74.5 per cent for the untreated fuel.

There is some doubt as to how far a comparison may be made between the operation and behavior of the laboratory-size producer used in this work and that of an actual industrial producer. Certainly the definite temperatures and gas compositions cannot be accepted as those to be found in large practical producers, but general trends should be discernible. With this in mind, a discussion of the advantages and disadvantages of adding sodium carbonate to the fuel bed may be made.

In some industrial operations the gases from the producer are piped to remote points before use. In this case much of the sensible heat of the gases, plus the latent heat of the steam that is condensed, is lost. Therefore, the temperature of operation should be as low as possible, with a high percentage of water decomposed, and a low carbon dioxide content in the exit gases. To meet these conditions, present producer practice calls for a temperature of approximately 1100° C. in the reaction zone, with an exit gas temperature of 800° to 850° C. Results of this investigation show that at 950° C. in the reaction zone, the percentage of steam decomposed by a fuel treated with sodium carbonate is between 85 and 100 per cent, and the dioxide concentration in exit gases is less than one per cent. The exit gas temperature is 700° to 750° C. Therefore, being able to operate at lower tempera-

tures the sensible heat losses would be reduced, and the heat losses from undecomposed steam would be negligible.

Many coals with characteristics desirable for use in producer practice cannot be used because of the low fusibility of the ash. In order to have high capacities and low operating trouble, coals whose fusion point of ash is approximately 1400° C. or higher should be employed. If the highest temperature in the producer, which is on or near the grate, could be brought below the temperature at which clinker formation begins, then coals of low ash fusibility could be used. A gas producer using a fuel treated with sodium carbonate should be able to operate with a grate temperature slightly higher than the temperature to be found in the reducing zone. Since the operating temperature of this reducing zone need not be higher than 950° C., the grate temperature would vary between 950° and 1000° C. Then coals whose fusibility of ash is approximately 1200° C. could be used without giving much clinker formation. This would permit the use of many coals that today are not favored in producer practice or, if they are used, would improve the ease of operation, thereby permitting higher capacities.

The present capacities or the average rate of gasification of coal in American practice is about 15 pounds per square foot of grate area per hour, although higher rates are possible when good coals are used. The depth of fuel bed ranges from 3 to 7 feet. The rates of gasification of graphite in the present work varied from 10 to 15 pounds per square foot of grate area per hour. The depth of the fuel bed was 10 to 12 inches. With deeper fuel beds, a higher rate of gasification would be possible because gas velocities could be increased without affecting the composition of the exit gases. A total time of contact of 0.3 second, which corresponded to a limiting velocity of air at which complete humidification took place in the bubble towers of the equipment used here, did not affect the composition of the exit gases of a fuel bed 10 inches deep and gave a rate of gasification of carbon of 25 pounds per square foot of grate area per hour.

If sodium carbonate were to combine with the excess siliceous ash of the fuel, the salt formed would be inert as far as the fuel bed is concerned and would pass out with the ashes. It is possible that the addition of calcium carbonate or lime would minimize this loss by forming a calcium instead of a sodium salt. The amount carried out of the producer in the exit gases has been negligible in this work, but it is not certain that it would be negligible at high rates of operation, especially if there was much dust carried out of the producer.

This question of the loss of soda in the ash and in the exit gases, and the degree of fluxing effect of the soda on the refractories must be studied further before the commercial possibilities of the process can be adjudged.

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